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Date/Time

Received for Laboratory by: (Signature)

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Relinquished by: (Signature)

Remarks

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Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 1 of 6 Lab Proj #: P0508313 Report Date: 09/06/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Laboratory Results

Total pages in data package:

Lab Sample #	Client Sample ID
P0508313-01	MW-17-5
P0508313-02	MW-17-4
P0508313-03	MW-17-3
P0508313-04	MW-17-2
P0508313-05	MW-17-1

Microseeps test results meet all the requirements of the NELAC standards.

Approved By:

The analytical results reported here are reliable and usable to the precision expressed in this report. As required by some regulating authorities, a full discussion of the uncertainty in our analytical results can be obtained at our web site or through customer service. Unless otherwise specified, all results are reported on a wet weight basis.

As a valued client we would appreciate your comments on our service.

Please call customer service at (412)826-5245 or email bhans@microseeps.com

Case Narrative:

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 2 of 6
Lab Proj #: P0508313
Report Date: 09/06/05
Client Proj Name: JPL Task 3 OU3
Client Proj #: G486111-T3

Sample Description MW-17-5	<u>Matrix</u> Water	<u>Lab Sample #</u> P0508313-01		mpled Date/Time 5 Aug. 05 9:30	Received 18 Aug. 05 12:	14
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	21.000	0.400	mg/L	AM20GAX	8/29/05	sl
TIC	220.0	4	mg/l CaCO	3 AM20GAX	8/29/05	rw

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 3 of 6
Lab Proj #: P0508313
Report Date: 09/06/05
Client Proj Name: JPL Task 3 OU3
Client Proj #: G486111-T3

Sample Description MW-17-4	<u>Matrix</u> Water	<u>Lab Sample #</u> P0508313-02	ALLEY JOHN	mpled Date/Time 5 Aug. 05 10:45	Received 18 Aug. 05 12:	14
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	22.000	0.400	mg/L	AM20GAX	8/29/05	sl
TIC	250.0	4	mg/l CaCO	3 AM20GAX	8/29/05	rw

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 4 of 6
Lab Proj #: P0508313
Report Date: 09/06/05
Client Proj Name: JPL Task 3 OU3
Client Proj #: G486111-T3

Sample Description MW-17-3	Matrix Water	Lab Sample # P0508313-03		ampled Date/Time 5 Aug. 05 12:45	<u>Received</u> 18 Aug. 05 12:14	
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	By
RiskAnalysis						
Nitrogen	18.000	0.400	mg/L	AM20GAX	8/29/05	sl
TIC	380.0	4	mg/I CaCC	3 AM20GAX	8/29/05	rw

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 5 of 6 Lab Proj #: P0508313 Report Date: 09/06/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description MW-17-2	<u>Matrix</u> Water	<u>Lab Sample #</u> P0508313-04		ampled Date/Time 15 Aug. 05 13:45	Received 18 Aug. 05 12:	4
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis Nitrogen TIC	19.000 450.0	0.400 4	mg/L mg/l CaC(AM20GAX O3 AM20GAX	8/29/05 8/29/05	sl rw

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 6 of 6
Lab Proj #: P0508313
Report Date: 09/06/05
Client Proj Name: JPL Task 3 OU3
Client Proj #: G486111-T3

Sample Description MW-17-1	<u>Matrix</u> Water	Lab Sample # P0508313-05		mpled Date/Time 5 Aug. 05 15:30	Received 18 Aug. 05 12:	14
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis Nitrogen TIC	21.000 250.0	0.400 4	mg/L mg/l CaCO	AM20GAX 3 AM20GAX	8/29/05 8/29/05	sl rw

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Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 1 of 6 Lab Proj #: P0506087 Report Date: 06/20/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Laboratory Results

Total pages in data package:

 Lab Sample #
 Client Sample ID

 P0506087-01
 Garfield

 P0506087-02
 Sunset

 P0506087-03
 Bangham

 P0506087-04
 LFWC-2

 P0506087-05
 LAWC-3

Microseeps test results meet all the requirements of the NELAC standards.

Approved By:

The analytical results reported here are reliable and usable to the precision expressed in this report. As required by some regulating authorities, a full discussion of the uncertainty in our analytical results can be obtained at our web site or through customer service. Unless otherwise specified, all results are reported on a wet weight basis.

As a valued client we would appreciate your comments on our service.

Please call customer service at (412)826-5245 or email bhans@microseeps.com

<u>Case Narrative:</u> The Inorganic Carbon is reported as dissolved. The TIC analyses for samples P0506087/01-03 were performed outside of the laboratory recommended holding time.

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 2 of 6 Lab Proj #: P0506087 Report Date: 06/20/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description Garfield	<u>Matrix</u> Water	<u>Lab Sample #</u> P0506087-01		Sampled Date/Time 02 Jun. 05 13:40	<u>Received</u> 06 Jun. 05 15:2	28
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	By
RiskAnalysis					······································	
Nitrogen	27.000	0.400	mg/L	AM20GAX	6/16/05	sl
TIC	210.0	4.0	ma/l	AM20GAX	6/17/05	sl

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 3 of 6 Lab Proj #: P0506087

Report Date: 06/20/05 Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
Sunset	Water	P0506087-02	02 Jun. 05 10:52	06 Jun. 05 15:28

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	22.000	0.400	mg/L	AM20GAX	6/16/05	sl
TIC	500.0	4.0	mg/l	AM20GAX	6/17/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 4 of 6 Lab Proj #: P0506087 Report Date: 06/20/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
Bangham	Water	P0506087-03	02 Jun. 05 15:30	06 Jun. 05 15:28

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	By
RiskAnalysis						
Nitrogen	24.000	0.400	mg/L	AM20GAX	6/16/05	sl
TIC	360.0	4.0	mg/l	AM20GAX	6/17/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 5 of 6 Lab Proj #: P0506087 Report Date: 06/20/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

 Sample Description
 Matrix
 Lab Sample #
 Sampled Date/Time
 Received

 LFWC-2
 Water
 P0506087-04
 03 Jun. 05 9:30
 06 Jun. 05 15:28

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis	04.000	0.400		AM000AV	CHCIDE	el
Nitrogen	24.000	0.400	mg/L	AM20GAX	6/16/05	SI .
TIC	360.0	4.0	ma/l	AM20GAX	6/17/05	Sl

Contact: Tien Shiao Address: 505 King Ave

TIC

Columbus, OH 43228

370.0

Page: Page 6 of 6 Lab Proj #: P0506087 Report Date: 06/20/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

AM20GAX

6/17/05

si

Sample Description LAWC-3	<u>Matrix</u> Water	<u>Lab Sample #</u> P0506087-05		Sampled Date/Time 03 Jun. 05 13:30	<u>Received</u> 06 Jun. 05 15:2	8
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis Nitrogen	25.000	0.400	mg/L	AM20GAX	6/16/05	sl

mg/l

4.0

Form No. 07/19/205

CHAIN OF CUSTODY RECORD

Baffelle POSO 334 Columbus Laboratories

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	Source Determination Study		SAMPLE I.D.	Mw-25-5	(X) h-52-mw	X X X X X X X X X X X X X X X X X X X	X	N-1-25-1			***				Date Time December 10: (Cinnettine)	60.	Date/Time Received by: (Signeture)	Date/Time Received for Laboratory by:
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Proj. No.	· 6-486 111-13	SAMPLERS:(Signature) \mathcal{L}_{\sim}	DATE	2/19/2005 F	2/19/1005	3002/61/F GO	•	057/19/2003								annewalco of the same of the s	Relinquished by: (Signature)	Relinquished by: (Signature)



Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 1 of 6 Lab Proj #: P0507334 Report Date: 08/02/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Laboratory Results

Total pages in data package: ___/

Lab Sample #	Client Sample ID
P0507334-01	MW-25-5
P0507334-02	MW-25-4
P0507334-03	MW-25-3
P0507334-04	MW-25-2
P0507334-05	MW-25-1

Microseeps test results meet all the requirements of the NELAC standards.

Approved By:

The analytical results reported here are reliable and usable to the precision expressed in this report. As required by some regulating authorities, a full discussion of the uncertainty in our analytical results can be obtained at our web site or through customer service. Unless otherwise specified, all results are reported on a wet weight basis.

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Please call customer service at (412)826-5245 or email bhans@microseeps.com

Case Narrative:

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 2 of 6
Lab Proj #: P0507334
Report Date: 08/02/05
Client Proj Name: JPL Task 3 OU3
Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-25-5	Water	P0507334-01	19 Jul. 05 12:30	21 Jul. 05 13:11

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	16.000	0.400	mg/L	AM20GAX	7/29/05	jl
TIC	280.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 3 of 6
Lab Proj #: P0507334
Report Date: 08/02/05
Client Proj Name: JPL Task 3 OU3

Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-25-4	Water	P0507334-02	19 Jul. 05 14:00	21 Jul. 05 13:11

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	33.000	0.400	mg/L	AM20GAX	7/29/05	jl
TIC	420.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 4 of 6 Lab Proj #: P0507334 Report Date: 08/02/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	Matrix	Lab Sample #	Sampled Date/Time	Received
MW-25-3	Water	P0507334-03	19 Jul. 05 15:10	21 Jul. 05 13:11

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	26.000	0.400	mg/L	AM20GAX	7/29/05	jl
TIC	370.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 5 of 6
Lab Proj #: P0507334
Report Date: 08/02/05
Client Proj Name: JPL Task 3 OU3

Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-25-2	Water	P0507334-04	19 Jul. 05 16:15	21 Jul. 05 13:11

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	27.000	0.400	mg/L	AM20GAX	7/29/05	jl
TIC	230.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 6 of 6 Lab Proj #: P0507334 Report Date: 08/02/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-25-1	Water	P0507334-05	19 Jul. 05 17:30	21 Jul. 05 13:11

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	26.000	0.400	mg/L	AM20GAX	7/29/05	jl
TIC	360.0	4.0	mg/l	AM20GAX	8/1/05	sl

Form No. 07/20, 2005

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CHAIN OF CUSTODY RECORD.

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To: Microserps Page 🚾

7/21/09 / 312

Date/Time

(Signature)

Date/Time

Relinquished by: (Signature)



Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 1 of 6 Lab Proj #: P0507335 Report Date: 08/02/05

Client Proj Name: JPL Task 3 OU3

Client Proj #: G486111-T3

Laboratory Results

Total pages in data package: ___/

Lab Sample #	Client Sample ID
P0507335-01	MW-19-5
P0507335-02	MW-19-4
P0507335-03	MW-19-3
P0507335-04	MW-19-2
P0507335-05	MW-19-1

Microse ps test results meet all the requirements of the NELAC standards.

Approved By:

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Please call customer service at (412)826-5245 or email bhans@microseeps.com

Case Narrative:

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 2 of 6 Lab Proj #: P0507335 Report Date: 08/02/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-19-5	Water	P0507335-01	20 Jul. 05 9:00	21 Jul. 05 13:16

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	16.000	0.400	mg/L	AM20GAX	7/29/05	jl
TIC	390.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 3 of 6 Lab Proj #: P0507335

Report Date: 08/02/05 Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description MW-19-4	<u>Matrix</u>	<u>Lab Sample #</u>	Sampled Date/Time	<u>Received</u>
	Water	P0507335-02	20 Jul. 05 11:00	21 Jul. 05 13:16

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	16.000	0.400	mg/L	AM20GAX	7/29/05	rw
TIC	390.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 4 of 6 Lab Proj #: P0507335 Report Date: 08/02/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

 Sample Description
 Matrix
 Lab Sample #
 Sampled Date/Time
 Received

 MW-19-3
 Water
 P0507335-03
 20 Jul. 05 12:10
 21 Jul. 05 13:16

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis	"					
Nitrogen	18.000	0.400	mg/L	AM20GAX	7/29/05	rw
TIC	370.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 5 of 6 Lab Proj #: P0507335 Report Date: 08/02/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample DescriptionMatrixLab Sample #Sampled Date/TimeReceivedMW-19-2WaterP0507335-0420 Jul. 05 14:0021 Jul. 05 13:16

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis Nitrogen	20.000	0.400	mg/L	AM20GAX	7/29/05	rw
TIC	280.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave

TIC

Columbus, OH 43228

420.0

Page: Page 6 of 6 Lab Proj #: P0507335 Report Date: 08/02/05

AM20GAX

8/1/05

sl

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description MW-19-1	<u>Matrix</u> Water	<u>Lab Sample #</u> P0507335-05		Sampled Date/Time 20 Jul. 05 15:00	<u>Received</u> 21 Jul. 05 13:10	6
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis Nitrogen	19.000	0.400	mg/L	AM20GAX	7/29/05	rw

mg/l

4.0

OSO 75 (S Form No. 07/21/ JOS

CHAIN OF CUSTODY RECORD

Baffelle

20516 1 to 186. in the field 404 Remarks 0/5954 Received by: Received by: (Signature) (Signature) Containers ło Number Container No. Date/Time Date/Time SAMPLE TYPE (V) Relinquished by: (Signature) Relinquished by: (Signature) X × × Received by: (Signature) Source Determination Study Received by: (Signature) 7-81-M SAMPLE I.D. MW-18-4 5-81-MW MW-18-3 1-81-MW 00: FI 2011/F Date/Time Date/Time **Project Title** 029 1500 1245 TIME 1030 2160 Relinquished by: (Signature) etinquisted by (Signature) SAMPLERS: (Signature) Columbus Laboratories G486111-73 50/12/E 50/17/6 50/12/6 7/21/05 7/21/05 DATE Proj. No.

Page of

To: Microsorps

Remarks

Date/Time

Received for Laborator by: (Signature)

Date/Time

Relinquished by: (Signature)



Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 1 of 6 Lab Proj #: P0507368 Report Date: 08/05/05

Client Proj Name: JPL Task 3 OU3

Client Proj #: G486111-T3

Laboratory Results

Total pages in data package: _____

Lab Sample #	Client Sample ID
P0507368-01	MW-18-5
P0507368-02	MW-18-4
P0507368-03	MW-18-3
P0507368-04	MW-18-2
P0507368-05	MW-18-1

Microseeps test results meet all the requirements of the NELAC standards.

Approved By:

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As a valued client we would appreciate your comments on our service.

Please call customer service at (412)826-5245 or email bhans@microseeps.com

Case Narrative:

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 2 of 6 Lab Proj #: P0507368 Report Date: 08/05/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

 Sample Description
 Matrix
 Lab Sample #
 Sampled Date/Time
 Received

 MW-18-5
 Water
 P0507368-01
 21 Jul. 05
 9:15
 22 Jul. 05
 15:17

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	21.000	0.400	mg/L	AM20GAX	8/3/05	sl
TIC	250.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave

Nitrogen

TIC

Columbus, OH 43228

Page: Page 3 of 6 Lab Proj #: P0507368

Report Date: 08/05/05 Client Proj Name: JPL Task 3 OU3

AM20GAX

Client Proj #: G486111-T3

sl

8/1/05

Sample Description MW-18-4	<u>Matrix</u> Water	<u>Lab Sample #</u> P0507368-02		Sampled Date/Time 21 Jul. 05 10:30	Received 22 Jul. 05 15:17	7
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis Nitrogen	22.000	0.400	mg/L	AM20GAX	8/3/05	sl

mg/L

mg/l

4.0

340.0

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 4 of 6 Lab Proj #: P0507368 Report Date: 08/05/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-18-3	Water	P0507368-03	21 Jul. 05 12:45	22 Jul. 05 15:17

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	15.000	0.400	mg/L	AM20GAX	8/3/05	sl
TIC	430.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 5 of 6 Lab Proj #: P0507368

Report Date: 08/05/05 Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-18-2	Water	P0507368-04	21 Jul. 05 16:30	22 Jul. 05 15:17

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	By
RiskAnalysis						
Nitrogen	18.000	0.400	mg/L	AM20GAX	8/3/05	sl
TIC	380.0	4.0	mg/l	AM20GAX	8/1/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 6 of 6 Lab Proj #: P0507368 Report Date: 08/05/05

Client Proj Name: JPL Task 3 OU3

Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-18-1	Water	P0507368-05	21 Jul. 05 15:00	22 Jul. 05 15:17

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	19.000	0.400	mg/L	AM20GAX	8/4/05	rw
TIC	270.0	4.0	mg/l	AM20GAX	8/1/05	sl

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in the lab.			<u></u>	×	1-1	MW-30-	1608	
please Filler			×	×	7-5	- 02 - CM	1407	
in the Field			×	×	6-0	Mw - 20		03 1-Aug-05
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Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 1 of 6 Lab Proj #: P0508066 Report Date: 08/16/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Laboratory Results

Total pages in data package: ___/

Lab Sample #	Client Sample ID
P0508066-01	MW-20-5
P0508066-02	MW-20-4
P0508066-03	MW-20-3
P0508066-04	MW-20-2
P0508066-05	MW-20-1

Microseeps test results preet all the requirements of the NELAC standards.

Approved By:

The analytical results reported here are reliable and usable to the precision expressed in this report. As required by some regulating authorities, a full discussion of the uncertainty in our analytical results can be obtained at our web site or through customer service. Unless otherwise specified, all results are reported on a wet weight basis.

As a valued client we would appreciate your comments on our service.

Please call customer service at (412)826-5245 or email bhans@microseeps.com

<u>Case Narrative:</u> The inorganic carbon samples were filtered in the lab according to the previously supplied instructions.

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 2 of 6 Lab Proj #: P0508066

Report Date: 08/16/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

 Sample Description
 Matrix
 Lab Sample #
 Sampled Date/Time
 Received

 MW-20-5
 Water
 P0508066-01
 01 Aug. 05
 8:57
 04 Aug. 05
 14:03

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	23.000	0.400	mg/L	AM20GAX	8/15/05	sl
TIC	260.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 3 of 6 Lab Proj #: P0508066

Report Date: 08/16/05 Client Proj Name: JPL Task 3 OU3

Client Proj #: G486111-T3

Sample Description MW-20-4	<u>Matrix</u> Water	Lab Sample P0508066-0		Sampled Date/Time 01 Aug. 05 10:50	<u>Received</u> 04 Aug. 05 14:	03
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	By
RiskAnalysis						
Nitrogen	21.000	0.400	mg/L	AM20GAX	8/15/05	sl
TIC	260.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 4 of 6 Lab Proj #: P0508066 Report Date: 08/16/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	Matrix	Lab Sample #	Sampled Date/Time	Received
MW-20-3	Water	P0508066-03	01 Aug. 05 12:30	04 Aug. 05 14:03

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis Programme RiskAnalysis						
Nitrogen	21.000	0.400	mg/L	AM20GAX	8/15/05	sl
TIC	430.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

TIC

Columbus, OH 43228

310.0

Page: Page 5 of 6 Lab Proj #: P0508066

Report Date: 08/16/05 Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

AM20GAX

8/7/05

Sample Description MW-20-2	<u>Matrix</u> Water	<u>Lab Sample</u> P0508066-0		Sampled Date/Time 01 Aug. 05 14:07	<u>Received</u> 04 Aug. 05 14:0	03
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	20.000	0.400	mg/L	AM20GAX	8/15/05	sl

4.0

mg/l

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 6 of 6 Lab Proj #: P0508066 Report Date: 08/16/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

A 1 - 4 - / - \	B	DOI.	Links Mathead #	Amelicaio Deta Di
MW-20-1	Water	P0508066-05	01 Aug. 05 16:08	04 Aug. 05 14:03
Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	BÀ
RiskAnalysis						
Nitrogen	18.000	0.400	mg/L	AM20GAX	8/15/05	sl
TIC	340.0	4.0	mg/l	AM20GAX	8/7/05	sl

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Form No.

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To: Mirroscops

1121 20/21/



Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 1 of 11 Lab Proj #: P0507426 Report Date: 08/08/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Laboratory Results

Total pages in data package:

Lab Sample #	Client Sample ID
P0507426-01	MW-24-4
P0507426-02	MW-24-3
P0507426-03	MW-24-2
P0507426-04	MW-24-1
P0507426-05	MW-24-5
P0507426-06	MW-21-5
P0507426-07	MW-21-4
P0507426-08	MW-21-3
P0507426-09	MW-21-2
P0507426-10	MW-21-1

Microseeps test results meet all the requirements of the NELAC standards.

Approved By:

The analytical results reported here are reliable and usable to the precision expressed in this report. As required by some regulating authorities, a full discussion of the uncertainty in our analytical results can be obtained at our web site or through customer service. Unless otherwise specified, all results are reported on a wet weight basis.

As a valued client we would appreciate your comments on our service.

Please call customer service at (412)826-5245 or email bhans@microseeps.com

Case Narrative:

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 2 of 11

Lab Proj #: P0507426 Report Date: 08/08/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description MW-24-4	<u>Matrix</u> Water	<u>Lab Sample</u> P0507426-		Sampled Date/Time 25 Jul. 05 9:00	Received 27 Jul. 05 12:1	2
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis Nitrogen TIC	18.000 220.0	0.400 4.0	mg/L mg/l	AM20GAX AM20GAX	8/6/05 8/7/05	mm sl

Contact: Tien Shiao Address: 505 King Ave Columbus, OH 43228

Page: Page 3 of 11 Lab Proj #: P0507426

Report Date: 08/08/05 Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description MW-24-3	<u>Matrix</u> Water	Lab Sample P0507426-0	-	Sampled Date/Time 25 Jul. 05 10:30	Received 27 Jul 05 12:1	2
Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis					The second secon	
Nitrogen	16.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	270.0	4.0	ma/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 4 of 11 Lab Proj #: P0507426 Report Date: 08/08/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

 Sample Description
 Matrix
 Lab Sample #
 Sampled Date/Time
 Received

 MW-24-2
 Water
 P0507426-03
 25 Jul. 05 12:00
 27 Jul. 05 12:12

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	18.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	290.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 5 of 11 Lab Proj #: P0507426
Report Date: 08/08/05
Client Proj Name: JPL Task 3 OU3
Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-24-1	Water	P0507426-04	25 Jul. 05 13:15	27 Jul. 05 12:12

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	By
RiskAnalysis						
Nitrogen	17.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	300.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 6 of 11 Lab Proj #: P0507426 Report Date: 08/08/05

Client Proj Name: JPL Task 3 OU3

Client Proj #: G486111-T3

 Sample Description
 Matrix
 Lab Sample #
 Sampled Date/Time
 Received

 MW-24-5
 Water
 P0507426-05
 25 Jul. 05 15:30
 27 Jul. 05 12:12

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	17.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	330.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 7 of 11 Lab Proj #: P0507426

Report Date: 08/08/05 Client Proj Name: JPL Task 3 OU3

Client Proj #: G486111-T3

 Sample Description
 Matrix
 Lab Sample #
 Sampled Date/Time
 Received

 MW-21-5
 Water
 P0507426-06
 26 Jul. 05 8:30
 27 Jul. 05 12:12

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	19.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	390.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 8 of 11 Lab Proj #: P0507426 Report Date: 08/08/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-21-4	Water	P0507426-07	26 Jul. 05 10:30	27 Jul. 05 12:12

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	18.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	400.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 9 of 11 Lab Proj #: P0507426

Report Date: 08/08/05 Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-21-3	Water	P0507426-08	26 Jul. 05 12:15	27 Jul. 05 12:12

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	21.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	590.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 10 of 11 Lab Proj #: P0507426 Report Date: 08/08/05

Report Date: 08/08/05
Client Proj Name: JPL Task 3 OU3
Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-21-2	Water	P0507426-09	26 Jul. 05 14:45	27 Jul. 05 12:12

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	By
RiskAnalysis						
Nitrogen	19.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	610.0	4.0	mg/l	AM20GAX	8/7/05	sl

Contact: Tien Shiao Address: 505 King Ave

Columbus, OH 43228

Page: Page 11 of 11 Lab Proj #: P0507426 Report Date: 08/08/05

Client Proj Name: JPL Task 3 OU3 Client Proj #: G486111-T3

Sample Description	<u>Matrix</u>	Lab Sample #	Sampled Date/Time	Received
MW-21-1	Water	P0507426-10	26 Jul. 05 16:00	27 Jul. 05 12:12

Analyte(s)	Result	PQL	Units	Method #	Analysis Date	Ву
RiskAnalysis						
Nitrogen	11.000	0.400	mg/L	AM20GAX	8/6/05	mm
TIC	340.0	4.0	mg/l	AM20GAX	8/7/05	sl

Microseeps, Inc.
SOP-AM20GAx In House Only Version
Revision: 4.0

Date: March 01, 2005 Page: 1 of 19

Microseeps, Incorporated

Analytical Method AM20GAx Standard Operating Procedure for the Analysis of Biodegradation Indicator Gases

Controlled Copy No. _____

Ruth Welsh

Laboratory Manager

Signature of Final Approval:

Patrick McLoughlin, Ph.D.

Technical Director

3-/-05

Date

SOP Review Date: March 1, 2005

Microseeps, Inc. SOP-AM20GAx In House Only Version Revision: 4.0

> Date: March 01, 2005 Page: 2 of 19

1.0 Scope and Application

Method AM20GAx is used to determine the concentration of biodegradation indicator gases in vapor samples. Specifically, Method AM20GAx is used to determine the dissolved concentration of the following gases:

Gases	CAS Number
Acetylene	74-86-2
Carbon dioxide	124-38-9
Oxygen	7782-44-7
Nitrogen	7727-37-9
Hydrogen	1333-74-0
Methane	74-82-8
Ethane	74-84-0
Ethene	74-85-1
Propane	74-98-6
Propene	115-07-1
n-Butane	106-97-8
i-Butane	75-28-5
Carbon Monoxide	630-08-0
Total Inorganic Carbon*	

^{*}Total inorganic carbon (TIC) is converted to carbon dioxide using the steps outlined in SOP-PM01. The sample is then analyzed for carbon dioxide according to this SOP. Any differences in method are specified in the appropriate section.

This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Method Summary

The sample gas is analyzed with a gas chromatograph capable of simultaneous analysis of all of the target analytes from a single 10 mL gas sample. A single injection of gas from integral, simultaneously filled sample loops is used to assure consistent injection volume. The permanent gases are analyzed using a thermal conductivity detector (TCD). The light hydrocarbons are analyzed using a flame ionization detector (FID). Hydrogen is analyzed using a reduction gas detector (RGD). The data are transferred to a microcomputer, converted to digital format, stored, and processed using a chromatography data system.

Revision: 4.0 Date: March 01, 2005

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2.1 Definitions

Batch: A sample batch consists of twenty or fewer samples run during an eight-hour work shift.

Instrument Flush: The front end of the sample loop is flushed with ultra high purity helium injected into the loop directly from the cylinder to remove possible interference by ambient air and to avoid cross contamination between samples.

Method Blank: A sample analyzed by all three detectors that consists of ultra high purity helium. The method blank is free from the analytes of interest

Laboratory Control Sample: A sample spiked with verified known amounts of analytes. A LCS is used to assess the performance of the measurement system.

Matrix Spike and Matrix Spike Duplicate: A sample prepared by adding a known concentration of target analyte to a specific amount of sample. Matrix spikes are used to determine the effect of sample matrix on a method's recovery efficiency.

3.0 Apparatus and Materials and Operating Conditions

3.1 Apparatus

Gas Chromatograph: The chromatographs designed and built by Microseeps are equipped with multiple packed columns and multi-port valves, a TCD, a FID, a RGD, and multiple sample loops. The FIDs, which were also built by Microseeps, are of a special design that allows considerably more sensitivity than commercially available models. To increase the working range of the system, there are two outputs to the FID. Thus, it is a four-channel system: (1) FID low; (2) TCD; (3) RGD; and (4) FID high. As discussed in Section 5.3, each channel is calibrated separately. This instrument provides rapid turn-around for consecutive analyses and a clean baseline for accurate, reproducible results.

3.1.1 Column Specifications

- Column 1: 80/100 mesh alumina packing material; 6' length, 3/16" OD; stainless steel, pre-washed (for hydrocarbon analysis).
- Column 2: 80/100 mesh Molesier 5A packing material; 12' length, 1/8" OD; stainless steel, pre-washed, preconditioned (for dissolved gas analysis).
- Column 3: 80/100 mesh Hayesep Q packing material; 12' length; 1/8" OD; stainless steel, pre-washed, preconditioned (for dissolved gas analysis).

Revision: 4.0 Date: March 01, 2005

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3.2 Materials

• Sample vials (Supelco, Inc, Bellefonte, PA or equivalent)

Syringe: locking gas tight

3.3 Operating Conditions

Gas Chromatograph:

Sample Loop Temperature: ambient
 Thermal Conductivity Detector Temperature: 100°C
 Flame Ionization Detector Temperature: ambient
 Reduction Gas Detector Temperature: 280°C

• Oven Temp.: 100 °C. isothermal

TCD Signal Range:

FID Signal Range: variable depending on concentrations

RGD Signal Range:

He Carrier Gas Regulator Pressure:
 60 psig

Sample carrier flow: 30 mL/min.
Reference flow: 30 mL/min.

N2 Carrier Gas Regulator Pressure: 25 psig
 Sample carrier flow: 25 mL/min

Valve Air Pressure: 60 psig.

3.3.1 Interferences

The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air as a contaminant will dramatically affect the results. The analyst must take great care to ensure that air is flushed from the gas tight syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. An unrestricted flow (Instrument flush) of pure carrier gas from a 10 psig source should be allowed to flow through each sample loop for 30 seconds prior to each analysis.

As required, the analyst should demonstrate the absence of carryover contamination by analysis of the contents of the sample loop when purged with carrier gas. This demonstration should be performed when carryover contamination is suspected (after high samples). In the event that 'ghost peaks' (peaks similar to previous sample) appear when a pure carrier gas sample is analyzed (method blank), measures should be taken to eliminate the carryover contamination.

Revision: 4.0 Date: March 01, 2005

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4.0 Reagents

- Helium (UHP Gas)
- Nitrogen (UHP Gas)
- · Certified Commercial Gas Standards
- Benzalkonium chloride (BAK) solution Prepared by dissolving 12.08 g into 1L DI water.
- Tri-sodium phosphate (TSP) purchased as the dodecahydrate

4.1 Standard Preparation Procedures

Calibration standards are prepared by using the procedures below:

4.1.1 Vial Preparation

Headspace vials used for instrument calibration standards for this method are prepared as follows:

- Crimp and cap each vial, with stopper septa.
- Evacuate each vial to vacuum below 100 milli torr.
- Flush each vial to atmospheric pressure with the vial preparation gas. The gas used depends upon the detector that is being calibrated and is specified in Table 4.1 below:

 Detector
 Vial Balance Gas
 Standard Mix Vendor

 FID
 Nitrogen
 Spectra

 TCD
 Helium
 Scotty

 RGD
 Nitrogen
 Spectra and Scotty

Table 4-1

4.1.1 Preparing Calibration Standards

Instrumentation is calibrated using dilutions of custom certified gas mixes. (Refer to Table 4.1.1 for the correct amounts of standard mix and vial preparation gas to inject into prepared vials.)

- Prepare the correct number of vials for the detector being calibrated.
- Inject the specified amount of standard by extracting it from the standard mix gas cylinder using a gas-tight syringe and injecting it into a prepared vial.
- Then the specified amount of vial balance gas is added to the same vial.

Date: March 01, 2005 Page: 6 of 19

The dilution factor of one is achieved by directly injecting the standard gas mix from the cylinder into the GC.

Table 4.1.1
Standard Gas and Balance Gas Injection Volumes in ml

Dilution Levels	Standard Gas Mix	Balance Gas	Final Gas Volume
1	N/A	N/A	N/A
2	21	21	42
5	10	40	50
10	5	45	50
25	2	48	50
100	2	198	200
250	1	249	250
2500	20 (of 250x)	180	200

4.1.2 Calibration Standard Concentrations

Calibration standards are made up in the following concentrations as specified in Tables 4.1.2 A, B, and C. The true values of the calibration standards vary slightly from cylinder to cylinder. The values below are very close approximations. All standards are prepared using 22 cc headspace vials with stopper septum or 160cc serum bottles.

Table 4.1.2 A FID Calibration In PPMV

Compound	1X	5X	25X	250X	2500X
Methane	500	100	20	2	0.2
Ethane	500	100	20	2	0.2
Ethene	500	100	20	2	0.2
Propane	500	100	20	2	0.2
Propene	500	100	20	2	0.2
n-Butane	500	100	20	2	0.2
i-Butane	500	100	20	2	0.2
Compound	1X	2X	10X	50X	250X
Acetylene	100	50	10	2	0.4

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Table 4.1.2 B
TCD Calibration
In PPMV

Compound	1X	2X	10X	25X	100X
Carbon Dioxide	150,000	75,000	15,000	6,000	1,500
Oxygen	70,000	35,000	7,000	2,800	700
Nitrogen	665,000	332,500	66,500	26,600	6650
Methane	45,000	22,500	4,500	1,800	450
Carbon Monoxide	70,000	35,000	7,000	2,800	700

Table 4.1.2 C RGD Calibration In PPMV

Compound	10X	25X	100X	250X	2500X
Hydrogen	50	20	5	2	0.2

4.2 Quality Control Sample Preparation

Quality control samples are prepared as indicated below.

4.2.1 Laboratory Control Sample (LCS) and LCS Duplicate (LCSD)

The LCS and LCSD are prepared at a mid-calibration range and are made from the same source as the matrix spike and spike duplicate. The type of LCS/LCSD depends upon the original matrix of the sample. For samples that arrive as vapors, the LCS/LCSD is injected as a gas. For samples that arrive as waters, DI water is spiked with a gas mixture of target analytes and prepared the same as the samples. Water that is free of the principle atmospheric components of nitrogen and oxygen is very difficult to make and similarly difficult to store. Toward that end, LCS/LCSD results for nitrogen will not be reported with client data. Table 4.2.2 below gives the true values of both the LCSs and MS/MSDs.

4.2.1.1 Total Inorganic Carbon LCS

Mix approximately 0.20g NaHCO₃ into 200ml H₂O, prepare according to the TIC procedures outlined in PM01 and analyze in duplicate as a sample. The true value of the spike is calculated as follows:

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Mg/L CaCO₃ =
$$\frac{Mass(g)NaHCO_3}{H_2O(L)} X \frac{100.09}{84.01} X (1,000,000)$$

4.2.2 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

MS and MSDs are prepared, analyzed, and reported when clients' request and send sufficient numbers of aliquots to prepare them (e.g. one 40 ml vial each for the MS and the MSD). They are prepared, one at a time, as follows:

- Using a clean 50ml gas-tight locking syringe, withdraw a volume of water from the bottom of the sample vial.
- Withdraw 10 cc of the certified standard gas used for preparing the LCSs and lock the syringe.
- Shake the syringe by hand (for use a wrist action shaker) for five minutes.
- The equilibrated MS and/or MSD is/are now ready to be analyzed.

Table 4.2.2 LCS/MS/MSD True Values

Compound	Vapor (ppmv)	Water
Methane	300	65.84 μg/L
Ethane	100	41,700 ng/L
Ethene	100	38,540 ng/L
Propane	100	60,560 ng/L
Propene	100	57,810 ng/L
iso-Butane	100	79,830 ng/L
n-Butane	100	79,830 ng/L
Carbon dioxide	50,000	30.22 mg/L
Oxygen	20,000	8.720 mg/L
Nitrogen	balance gas	balance gas
Hydrogen	25	344.1 nM

Notes on Table 4.2.2

- MS/MSD not performed on vapor samples and results are corrected for water samples.
- Actual values vary slightly from lot to lot of cylinders of calibration gases.
- MS/MSD prepared by using 10cc of standard instead of 10cc He in the headspace prep. procedure.

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4.2.2.1 Total Inorganic Carbon MS and MSD

Mix approximately 0.04g NaHCO₃ directly into client samples (when provided and requested), prepare according to the TIC procedures outlined in PM01 and analyze in duplicate as a sample. The true value of the spike is calculated as follows:

Mg/L CaCO₃ =
$$\frac{Mass(g)NaHCO_3}{H_2O(L)} X \frac{100.09}{84.01} X (1,000,000)$$

4.2.3 Method Blank

Method blanks are made up of ultra high purity helium injected into a vial and then into the instrument.

4.2.3.1 Total Inorganic Carbon Method Blank

The method blank for TIC is made up of deionized water in a 40 ml vial, prepared according to the TIC procedures outlined in PM01, and analyzed as a sample.

4.3 Glassware and Storage Requirements for Reagents and Standards

Reagents are stored at room temperature (70°F \pm 5°) and all standards are prepared fresh for each use immediately prior to each analysis. Standards are made up from compressed gas cylinders. Those standards expire after 2 years.

5.0 Procedure

Water samples should be cooled upon shipment and stored at a temperature of $4^{\circ}\text{C} \pm 2^{\circ}$. Gas samples are shipped and received at a positive pressure, which eliminates a cross-contamination issue during sample shipment. It is preferable that gas samples be shipped without cooling. However, it is not a sample receipt non-conformance if received packed in ice (sample may experience slight loss in pressure.) Gas samples are stored in the laboratory at room temperature (70°F $\pm 5^{\circ}$). The pressure in gas vials is not checked upon receipt in the laboratory because of the inherent risk of losing sample, or inadvertently introducing atmospheric gases, when the septum is pierced. The number of times the septum is pierced should be as few as absolutely possible. See Section 5.2.2 for a discussion on how the laboratory checks and documents vial pressure. Holding time for both gas and water samples is fourteen days.

Water samples for light hydrocarbon analyses only (methane, ethane, ethane, propane, propene, n-butane, i-butane, acetylene) are collected in 40ml VOA vials with zero headspace and preserved with tri-sodium phosphate (TSP). TSP is added as the dodecahydrate at 200 mg/40 ml vial. This results in a sample pH > 10. Water samples collected for either permanent gases only

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or permanent gases and light hydrocarbon analyses are collected in 40ml amber VOA vials with zero headspace and preserved with four drops of BAK solution.

Analysts who use this method have been certified for the method by running Initial Demonstration of Proficiency (IDOP) Samples in accordance with Microseeps Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and Instrumentation (SOP ADM 02). IDOPs are run any time there is significant change to an instrument, method, or in the training procedure for training a new analyst.

5.1 Sample Preparation

Samples that are collected using the Bubble Strip Sampling Technique, Microseeps Sampling Method SM9, do not require additional preparation prior to analysis.

Samples that are collected as waters and are to be analyzed for dissolved gases (methane, ethane, ethene, acetylene, CO₂, N₂, O₂, propane, propene, iso-butane, n-butane, TIC), must be prepared using Microseeps Standard Operating Procedure PM01G.

Samples that are collected as gases, for example from a soil gas survey or from the headspace of a microcosm sample, need not be collected by a Microseeps sampling method, nor do they require additional preparation.

5.2 Analysis

- **5.2.1** If the sample is prepared via SOP-PM 01, it can be injected from the gastight syringe in which it is prepared by inserting the needle of the syringe through the septum on the "sample in" port. If the sample is a calibration standard, a bubble strip sample (SM9), or a gas, the septum inlet to the "sample in" port of the GC must be removed and a luer-lock needle receptacle is plumbed to the "sample in" port in place of the needle. A needle is attached to the luer-lock receptacle and inserted through the septa of the calibration standard, bubble stripped sample, or gas sample.
- **5.2.2** In order to initiate analysis and introduce the sample into the GC sample loop, a needle is attached to the entry port on the GC and inserted through the sample septum. The flow through the sample loop is monitored by a flow meter connected to the sample-loop vent-port on the gas chromatograph.

When a vial is sufficiently filled, the ball in the flow meter will shoot to the top of the column. This indicates that there is sufficient pressure in the vial to fill the sample loop. If the loop is not properly pressurized, this is reflected on the flow meter immediately. The ball in the flow meter will go up the column part way and drop back to the bottom. This indicates there is not sufficient pressure in the sample vial. If this happens, the analyst will remove the vial from the inlet port as quickly as possible and withdraw $10 - 12\cos$ of sample from the sample vial using a locking

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syringe. This is then injected into the instrument. The lack of sufficient pressure in the vial and the means of sample injection are then documented on the case narrative.

- **5.2.3** Once the flow out of the sample loop ceases (3 seconds if SOP-PM 01 is used) the sample loop valves are activated.
- **5.2.4** Once the sample loop valves are activated, the ports to and from the sample loop are flushed with ultra high purity helium injected into the loop directly from the cylinder to remove any interference from ambient air and to avoid cross contamination between samples.

5.3 Calibration and Results

5.3.1 The standard calibration gas should be introduced in the same manner as described in section 5.2.1 above. Measured peak areas are converted to concentrations using certified commercial gas standards. Dilutions are made to achieve multi-point calibration curves for each detector.

Methane can be detected on both the FID and the TCD. If the methane concentration causes an FID signal output level of 8000 millivolts, then any output exceeding that is quantified on the TCD.

5.3.2 Initial calibration is accomplished by analyzing multiple standards of appropriate calibration ranges.

Note: Due to the nature of preparing custom gas standards, the component concentration can fluctuate between purchased lots. This is accounted for during method/calibration development. These results will be used to establish a multi-point calibration curve.

Acceptance Criteria: A linear fit to an area response versus concentration plot is formed with the origin forced to zero, and the calibration is accepted for use if r^2 , the coefficient of determination is ≥ 0.995 .

Corrective Action: If the acceptance criteria specified above is not met, the reason is determined and a new set of calibration standards are analyzed.

5.3.3 An Initial Calibration Verification (ICV) standard immediately follows the initial calibration. The ICV is made up from a second source and is identical to the LCS used for the analysis of vapors. Acceptance criterion for the ICV is an instrument response of \leq (less than or equal to) 20% (%D).

Acceptance Criteria and Corrective Action: If the instrument response for the ICV standard varies by more than 20% (%D), the analyst will not analyze samples until, either the reason is determined and the problem is corrected, or a new multi-point calibration is analyzed.

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5.3.4 An initial calibration blank follows the ICV. The blank is made up of the carrier gas. Compounds must not be detected above the reporting limits.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.3.5 The analytes of this method are indicators. Every attempt to achieve and deliver precise results is made. However, it is realized that for indicator parameters measuring the range of the analyte concentration (i.e. is the concentration of methane gas >1 mg/l or < 0.1 mg/l) is the primary goal of employing these analyses. The calibration range is chosen to extend over most of the bio-indicator concentration range. If the concentration of an analyte exceeds that of the highest calibration standard, but does not saturate the instrument response, the concentration is calculated by assuming detector response linearity and using an extrapolation of the calibration plot. If the instrument response is saturated the sample is diluted to bring the analyte concentration into the calibration range.

5.4 Quality Control

The following quality control samples shall be analyzed with each analytical batch of twenty or fewer samples.

5.4.1 A Continuing Calibration Verification: The CCV is made up from a source other than what was used to make up the initial calibration. The acceptance criterion for the CCV is a percent drift of $\pm 20\%$.

Corrective Action: If the CCV fails, the instrument shall be recalibrated, and all samples since the last acceptable calibration shall be reanalyzed, provided sufficient sample volume is present and the samples have not been compromised by exposure to air.

5.4.2 A Continuing Calibration Blank: A CCB follows each CCV. The blanks are made up of the carrier gas. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.3 Laboratory Control Sample and Laboratory Control Sample Duplicate: The LCS and LCSD are prepared and analyzed at a mid-calibration range.

Acceptance Criteria: Percent recovery is required to be between 75% and 125%, inclusive. Acceptance criterion is based upon the percent recovery and the RPD as calculated by:

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$$Percent \, Re \, cov \, ery = \frac{Measured Value}{True Value} \times 100\%$$

$$RPD = \frac{|C1 - C2|}{\frac{C1 + C2}{2}} \times 100\%$$

Where: C1=LCS C2=LCSD

RPD (Relative Percent Difference) is required to be less than or equal to 20%.

Corrective Action: If the LCS fails, a new LCS is prepared and analyzed. If the new LCS falls within the acceptance criterion, analysis continues. If the new LCS fails, analysis is stopped and the instrument is checked with a series of standards to determine the cause. Once the cause is determined and the instrument repaired, calibration is conducted and analysis continues.

5.4.4 Matrix Spike and Matrix Spike Duplicate: Matrix spikes and spike duplicates are analyzed for water samples only when requested by a client and sufficient sample aliquots are provided. Acceptance criterion is a percent recovery between 70% and 130%, and a relative percent difference of less than or equal to 20%.

Corrective Action: If the matrix spike and spike duplicate fail but all the other quality control samples are within the acceptance criteria, matrix interference is noted in the Case Narrative.

5.4.5 Method Blank: A method blank is analyzed with each sample batch. The blanks are made up of UHP helium for all of the gases except for blanks for TIC. TIC blanks are made up of deionized water. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.6 Contingency for Handling Out of Control or Unacceptable Data

If the requirements set forth in section 5.4 are not met, the analytical program will be terminated until the cause is determined and a solution is affected. All samples associated with out of control quality control samples (with the exception of matrix interference) must be reanalyzed provided another vial of sample has been provided by the client. If quality control acceptance criteria cannot be met using the corrective action above, a detailed check of the analytical system is made. Reagents, standards, and other quality control samples are re-prepared and analyzed. If problems persist, sample analysis will be halted and the Laboratory Manager shall be contacted immediately to determine the cause and implement corrective action.

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Any data submitted with unacceptable quality control sample results shall be qualified in a case narrative. The narrative should indicate the out of control event that occurred, the corrective action that was taken, and any other pertinent information to inform the client of exactly what occurred.

- 5.4.7 An experienced analyst shall examine all chromatograms.
- **5.4.8** Through out analysis the gas samples are injected mechanically into the GC flow path utilizing a sample loop to achieve a uniform sample size from a flow directly from the sample preparation syringe. The uniform sample size achieved using the sample loop assures consistent and accurate results. Table 5.4.8 (see next page) gives example data from a study performed via this analysis. That data can also be used for accuracy and precision estimates.

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Table 5.4.8 Example Data for Precision and Accuracy Studies

	Carbon	_	Nitrogen	Markone	11.dana	Machan	1	1,1				
	Dioxide	Dioxide Oxygen	Ingo livi	Memane	riyarogen	Memane	Emane	Ethylene	Propane	Propylene	Iso-Butane N-Butane	N-Butane
REP.#	(%)	(%n)	(%v)	(%)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)
	0.1221	0.0670	0.5744	0.0410	0.1118	0.2512	0.0525	0.0453	0.0461	0.0581	0.0473	0.0358
2	0.1267	0.0690	0.6020	0.0428	0.1122	0.2608	0.0518	0.0468	0.0521	0.0465	0.0439	0.0407
3	0.1207	0.0657	0.5838	0.0446	0.1247	0.2812	0.0509	0.0485	0.0529	0.0588	0.0436	0.0405
4	0.1193	0.0667	0.6036	0.0444	0.1244	0.2779	0.0549	0.0460	0.0461	0.0536	0.0549	0.0476
5	0.1261	0.0703	0.5860	0.0439	0.1120	0.2894	0.0551	0.0497	0.0520	0.0549	0.0417	0.0460
9	0.1193	0.0665	0.5861	0.0478	0.0943	0.2970	0.0515	0.0467	0.0458	0.0542	0.0435	0.0514
7	0.1227	0.0732	0.5748	0.0353	0.1296	0.3053	0.0532	0.0473	0.0485	0.0584	0.0483	0.0535
AVERAGE	0.1224	0.0683	0.5872	0.0428	0.1156	0.2804	0.0528	0.0472	0.0491	0.0549	0.0462	0.0451
KNOWN	0.1500	0.0700	0.6649	0.0450	6660.0	0.1500	0.0500	0.0500	0.0500	0.0500	0.0500	0.0500
STD. DEV.	0.003	0.003	0.012	0.004	0.012	0.019	0.007	0.001	0.003	0.004	0.004	9000

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5.4.9 The gas matrix for this analysis minimizes the opportunity for matrix effects. If the gas is prepared from a matrix other than that which is injected into the GC (e.g. prepared through headspace extraction via Microseeps SOP-PM01), the client should request that matrix spike (MS) and matrix spike duplicate (MSD) analyses be conducted and should supply sufficient sample volume. Since matrix effects are extremely site dependent, the MS and MSD are not part of the regular analytical quality assurance program.

5.4.10 All of the target analytes are gases at room temperature so the opportunity for carry over is negligible. Because of the configuration of the GC used in this analysis, any possible carryover would only manifest itself as a ghost peak, well out of the RT windows of any analytes and thus never misinterpreted. For these reasons, samples that have high concentrations of analytes do not need to be followed by a blank analysis.

5.5 Capturing and Submitting Data

The output of the chromatograph is directed to a microcomputer where the signal is converted to digital format, stored, and processed using a chromatography data system.

Automated valve control: Digital control is provided by the microcomputer though the chromatography data-system software. This control provides constant start and stop times for directing carrier gas flow. The event times are programmed and saved using the method editor module of the software.

5.5.1 Total Inorganic Carbon Result Calculation

The total inorganic carbon result is calculated as follows:

TIC as mg/L CaCO₃= (%CO₂)((Volume headspace)(2.08)+43.3)

This analysis produces concentration of the analyzed gas in PPMV or % V. If the sample was collected via the bubble-strip method (Microseeps SOP SM9) or prepared through static headspace preparation (Microseeps SOP DGPM 01), the gas phase concentrations can be used to specify sample water concentrations via the calculations presented in those Standard Operating Procedures.

5.5.2 Retention Time Windows

Retention time studies have been conducted for this analysis. These studies are kept on file in the Quality Systems Office. The retention times in Table 5.5.2 below are examples. The exact retention times will vary as a function of column type, column age, and column history. For the instruments that use this method, true retention times and retention time windows are taken from the most recent retention time window study conducted.

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Table 5.5.2 Retention Time Windows

Compound	RT Window (Min.)	RT Window (Min.)	RT Window (Min.)	RT Window (Min.)
	BioRen	a I Unit	BioRem	II Unit
Carbon Dioxide	5.171	5.340	4.058	4.635
Oxygen	6.537	7.015	5.686	5.721
Nitrogen	7.200	7.626	6.510	6.570
Methane	9.523	9.933	8.874	8.999
Carbon Monoxide	10.475	10.841	10.938	11.302
Methane	0.586	0.609	0.420	0.420
Ethane	0.809	0.835	0.730	0.730
Ethene	1.027	1.050	1.029	1.064
Propane	1.545	1.570	1.871	1.962
Propene	2.822	2.850	3.942	4.225
iso-Butane	3.763	3.807	5.804	6.230
n-Butane	4.351	4.399	6.855	7.379
Hydrogen	4.404	4.480	NA	NA

6.0 Secondary Data Review

All analytical data must undergo a minimum of a two-tiered review. The analyst first reviews the data for completeness and accuracy. The data is then submitted to the Group Lead Analyst for final review and the data is entered into the LIMS. Once approved at this level, the data is released as a final report.

7.0 Reporting Limits

The reporting limits for this analysis are listed in Table 7.0 below. Method detection limit studies are run annually in accordance with Microseeps Standard Operating Procedure for the Determination of Method Detection Limits and PQLs (SOP-ADM 18).

Those MDLs must be less than the reporting limits specified below. MDL studies are also performed when there is reason to suspect that method sensitivity has changed. The MDL studies are kept on file in the Quality Systems Office.

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Reporting Limits Table 7.0

Parameter	Reporting Limit	Units
Carbon Dioxide	0.02	%V
Oxygen	0.02	%V
Nitrogen	0.04	%V
Hydrogen	0.02	ppmv
Acetylene	0.34	ppmv
Methane	0.06	ppmv
Ethane	0.01	ppmv
Ethene	0.01	ppmv
Propane	0.01	ppmv
Propene	0.01	ppmv
n-butane	0.01	ppmv
i-butane	0.02	ppmv
Acetylene	500	ng/L

7.1 Conversion Factors

This procedure is used to measure the volume concentration of the analytes in a gas. Two methods are used to extract that gas from the groundwater. The conversion factors that are used to convert the concentration of the analytes in the water from the concentration of the analytes as they are measured using this method, are specific to the collection or preparation method and can be found in either SOP-SM9 or SOP-PM 01.

8.0 Safety

Gloves, proper eye protection, and a laboratory coat shall be worn when handling samples and standards. The major hazard in this laboratory area is stick from needles. All needles must be capped when not in use and when moving about the laboratory. The proper way of capping a needle is to place the cap on the laboratory bench and direct the needle into the cap. A needle is never to be directed into a cap while the cap is being held.

All compressed gases are to be moved using a dolly made for transporting gases and shall be chained in place when in the laboratory. The chain shall be tightened sufficiently to keep the cylinder upright if jostled.

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9.0 Laboratory Waste

Samples are kept for 30 days following analysis. Samples are disposed according to Microseeps Standard Operation Procedure for Waste Disposal (SOP-ADM 14).

9.1 Waste Minimization

Where possible, Microseeps takes steps to minimize the amount of waste generated in the laboratory by using substitution, where possible, and good chemical handling procedures. For specific information on waste minimization consult SOP-ADM 14.

10.0 References

Citing a reference does not imply that all of the recommendations and/or requirements in those cited methods is required in this Standard Operating Procedure. This section simply refers to sources that were consulted to gather information or knowledge in order to write an informed technical procedure.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste. SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC. 1986.

Newel, B.S., RSKSOP-175, <u>Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC Headspace Equilibration Technique</u>. Revision No. 0, August 1994.

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American Society for Testing and Materials, Standard Practice for Analysis of Reformed Gas by Gas Chromatography. <u>Annual Book of ASTM Standards</u>. Vol. 14.02, 1994.

Kampbell, D.H. and Vandegrift, S.A., Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique. <u>Journal of Chromatographic Science</u>. Vol. 36, May 1998.